

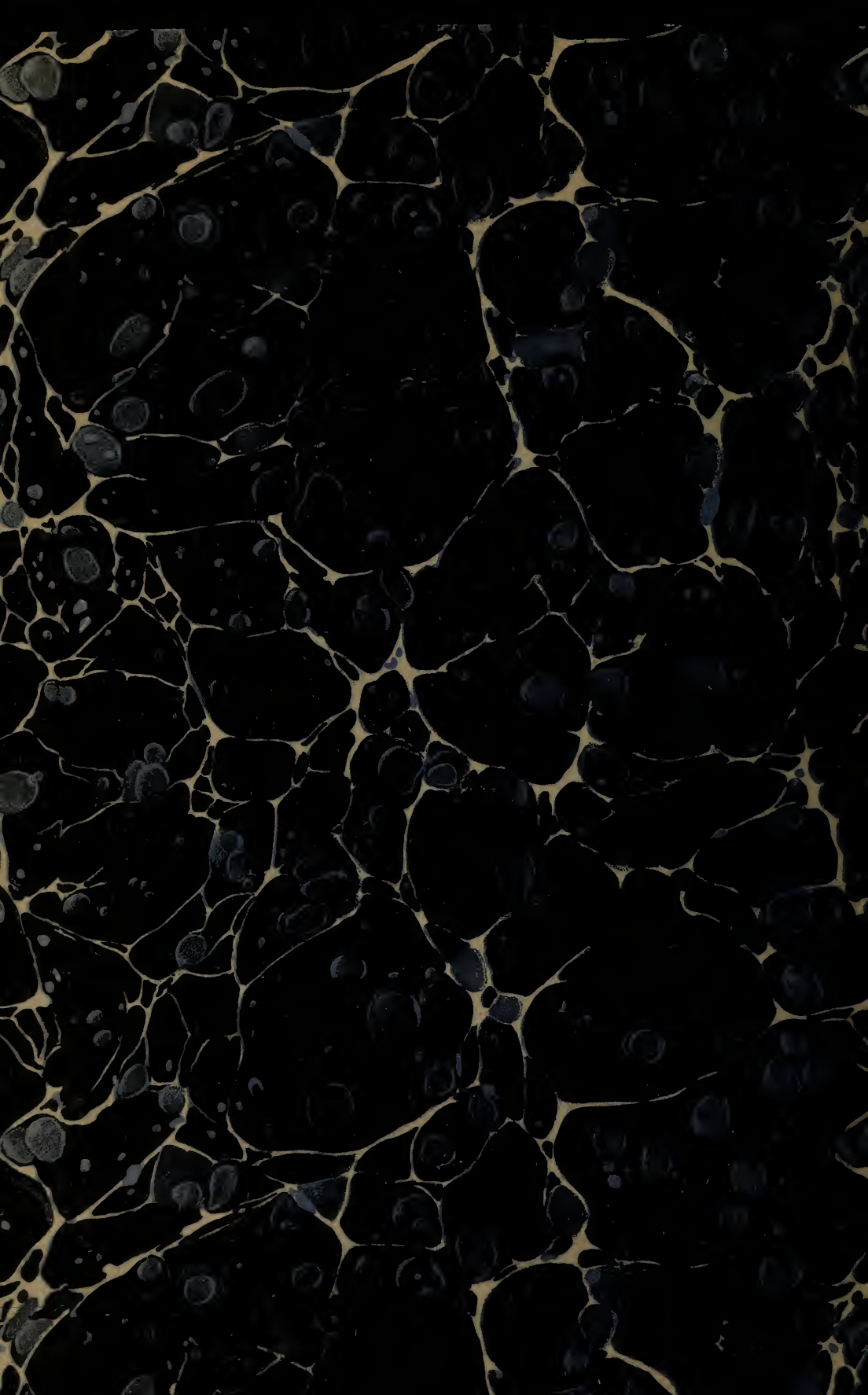
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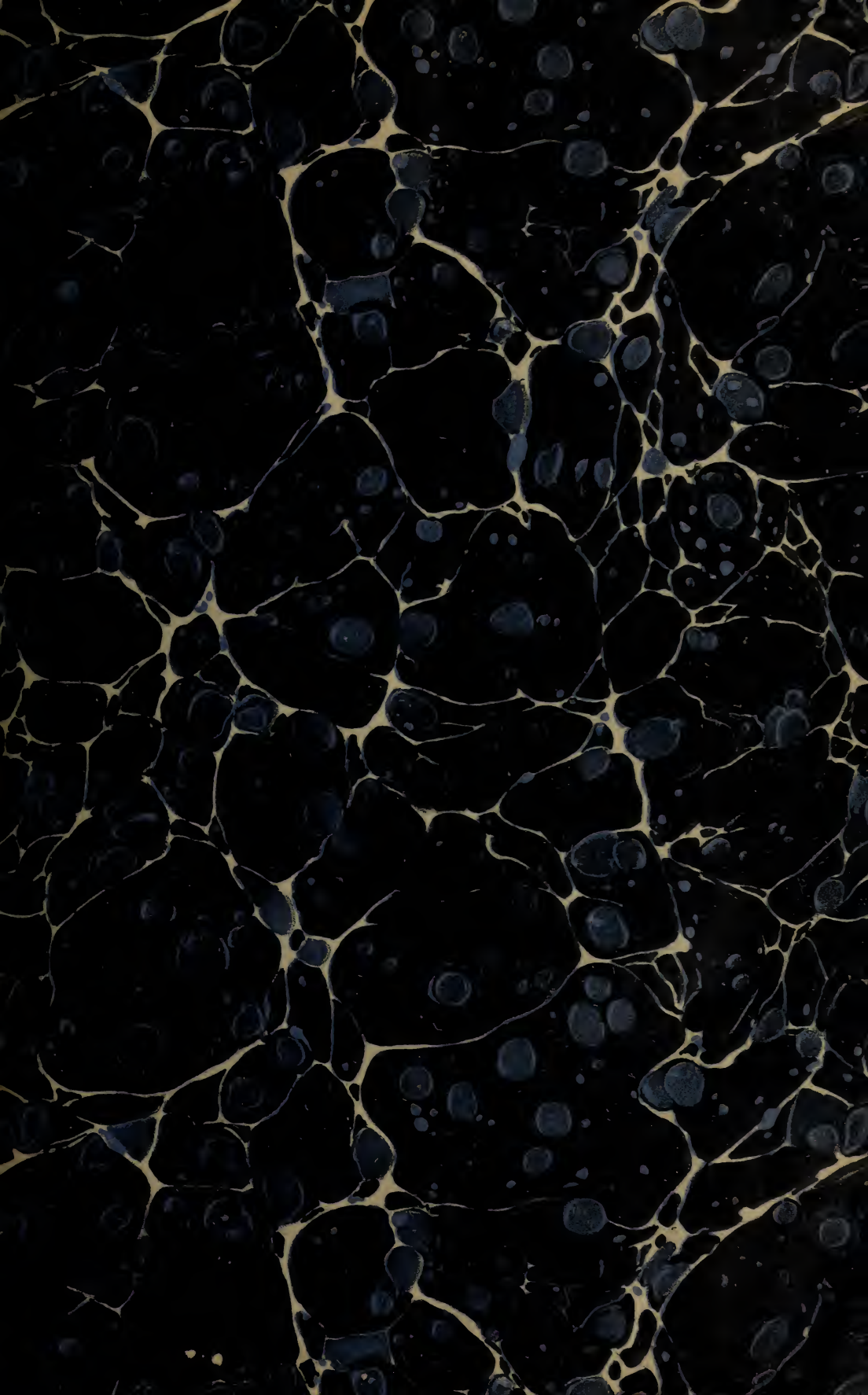
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TECHNOLOGIC PAPERS

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BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 62

MODERN PRACTICE IN THE CONSTRUCTION AND
MAINTENANCE OF RAIL JOINTS AND
BONDS IN ELECTRIC RAILWAYS

BY

E. R. SHEPARD, Associate Electrical Engineer

Bureau of Standards

ISSUED MARCH 10, 1916



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1916

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(Continued on page 3 of cover)

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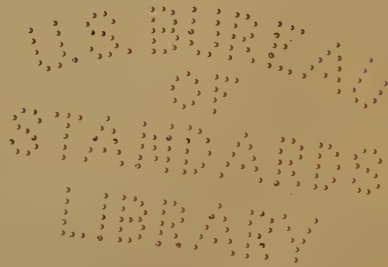
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SOME QUALITATIVE TESTS FOR GUM ARABIC AND ITS QUANTITATIVE DETERMINATION

By C. E. Waters and J. B. Tuttle, Associate Chemists

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I. INTRODUCTION

The group of polysaccharides includes such diverse substances as the starches, cellulose, the dextrins, the true gums, and the plant mucilages. They possess in common the property of being decomposable hydrolytically into one or more sugars, usually pentoses or hexoses. By oxidation, the acids corresponding to

these sugars are formed, but the first step toward this reaction appears to be hydrolysis.

The true gums, of which gum arabic is typical, dissolve in cold water, yielding clear solutions which, though viscous and adhesive, can be filtered. The closely related and quite similar mucilages, such as gum tragacanth and cherry-tree gum, simply swell up and form more or less homogeneous suspensions that can not be filtered. Perhaps most of the so-called gums are mixtures of one or more representatives of each of the above two classes.

As might be expected from their chemical nature, the gums do not readily lend themselves to reactions of a definite qualitative or quantitative value. One of us learned how unsatisfactory some of the qualitative tests are when samples of mucilage first came to the Bureau of Standards for examination. This led to an extended study of the literature and of the various qualitative reactions that have been published, and finally to a comparatively accurate quantitative method. In the last part of this work the two of us joined forces, hoping to carry on a much more extended investigation than was found later to be possible.

The object of this paper is to bring together all the more important qualitative and quantitative methods that have been published and to present a new procedure for the determination of gum arabic. Since in most cases it is necessary to know only whether gum arabic is present or not, it is believed that the following summary and criticism of the qualitative tests will be of value. Many of the quantitative methods depend on the same reactions.

II. QUALITATIVE TESTS

In nearly all cases a 2 per cent solution of gum arabic was used. Similar solutions of dextrin and of gum ghatti, a substitute for gum arabic, were subjected to the same tests.

1. FERRIC CHLORIDE AND ALCOHOL.—A solution of ferric chloride, made up without the addition of hydrochloric acid and filtered to remove a small amount of basic salt, was used. It contained about 25 g of the salt in 100 cc. Neither gum arabic, gum ghatti, nor dextrin gave a precipitate with this reagent.

A mixture of $2\frac{1}{2}$ volumes of 50 per cent alcohol and the ferric-chloride solution precipitates gum arabic, though often only on

long standing.¹ Gum ghatti gives no precipitate, and dextrin a very slight one.

2. POTASSIUM HYDROXIDE.—According to Liebermann,² solutions of gum arabic and of dextrin become amber-yellow when warmed with potassium hydroxide, while the closely related gum senegal gives at most a faint yellow color. Sollman³ stated that dextrin, when so treated, turns more or less brown, while some sugars and gums, other than arabic, behave similarly. Rideal and Youle⁴ came to the conclusion that this test is of no value. Two samples of gum arabic, among a number tested by them, gave a green color. They found that a solution of ghatti turns pink and dextrin a very dark red or almost black. Nevertheless, this test is among those recommended in the latest edition of Lunge.⁵

Our experiments with gums and sugars amply confirmed the statement that this test is of no value.

3. COLOR REACTIONS WITH PHENOLS.—Reiche⁶ found that gum arabic gave a flocculent blue precipitate when boiled with orcinol and concentrated hydrochloric acid. Other carbohydrates, including dextrin, cellulose, sugars, starch, and a number of gums, gave yellow or brown colors.

Ihl⁷ was able to distinguish between gum arabic, dextrin, and starch by means of α - and β -naphthol and phloroglucinol. With resorcinol and pyrogallol, all three carbohydrates gave the same color.

In this connection may be mentioned a curious reaction discovered by Clermont and Chautard.⁸ A solution of pyrogallol, containing gum arabic, gave a yield of 67 per cent of the condensation product, purpurogallin (pyrogalloquinone), on standing for two months.

In the hope of obtaining definite color reactions, hydrochloric acid-alcoholic solutions of the following phenols were made up: Phenol, resorcinol, pyrocatechol, guaiacol, hydroquinol, pyrogal-

¹ Roussin: *J. Pharm. Chim.*, [4], 7, p. 251; 1868. See also Allen, *Coml. Org. Anal.*, 4th ed., 3, p. 443.

² *Chem.-Ztg.*, 14, p. 665; 1890.

³ *Am. J. Pharm.*, 83, p. 176; *Chem. Zentr.*, 82, 1, p. 1560; 1911.

⁴ *J. Soc. Chem. Ind.*, 10, p. 610; 1891.

⁵ *Chem.-Techn. Untersuchungsmeth.*, 6th ed., 3, pp. 167-168; 1911.

⁶ *Ber. Ges. Förder. chem. Ind.*, 1879, p. 74; *Chem.-Ztg.*, 4, p. 191; 1880.

⁷ *Chem.-Ztg.*, 9, p. 231; 1885.

⁸ *Compt. Rend.*, 94, p. 1254; *Jahresber.*, 1882, p. 684.

lol, phloroglucinol, and α - and β -naphthol. These were heated, in test tubes set in beakers of water on the steam bath, with dextrin and gums arabic, ghatti, and tragacanth. A considerable range of colors resulted: Pale yellow, pale pink, red-brown, brown, pale orange, purple-red, etc., but no reliance could be placed in any of the reactions as a means of identification.

Some interesting results were obtained by heating the same carbohydrates with alcohol and hydrochloric acid, without the addition of a phenol. Dextrin gave no color, gum arabic a pale yellow, and tragacanth a pale pinkish-yellow. A sample of gum ghatti, prepared by powdering together a number of lumps taken at random, gave a pale-yellow solution with perhaps a tinge of pink. The addition of a clear lump of the gum caused no change, while a dark, impure piece produced a dark pink. There may be some connection between these color reactions and the behavior of different lumps of ghatti toward the guaiacol-hydrogen peroxide reagent, which is discussed further on.

4. BASIC LEAD ACETATE.—An aqueous solution of neutral lead acetate precipitates neither dextrin nor gums arabic and ghatti. An alcoholic solution of the salt does, however, slowly precipitate gum arabic.⁹

As might be expected from its employment in clarifying sugar solutions before polarization, basic lead acetate is an excellent precipitant for many gums. The solution may be made basic either by adding ammonia,¹⁰ or by boiling with litharge. The precipitate formed when gum-arabic solution is added is of a peculiar curdy consistency, and of such characteristic appearance that it is hardly necessary to make any confirmatory tests.

Allen¹¹ speaks of this precipitate as a "white jelly." Dextrin solutions are, at most, made slightly cloudy by the reagent, while gum ghatti yields a very small amount of precipitate, most of which remains suspended in the liquid.

The basic acetate solution used by us was made by long-continued boiling of 40 g of crystallized lead acetate, dissolved in 250 cc of water, with an excess of litharge. It was filtered, after which it remained clear for a long time.

⁹ Chauvin: *Mon. Sci.*, [5], 1, pp. 317-8; 1911; *Chem. Zentr.*, 82, 1, p. 1656; 1911.

¹⁰ v. Lippmann: *Chemie der Zuckerarten*, III Aufl., p. 1616.

¹¹ *Loc. cit.*

Basic lead acetate has been recommended probably more frequently than any other reagent as a qualitative test for gum arabic, as well as for removing this and other gums from wines, liqueurs, etc., in which other constituents are to be determined.¹²

5. COPPER SULPHATE AND SODIUM HYDROXIDE.—Jettel¹³ does not mention the basic lead acetate test, but relies chiefly upon the behavior of dextrin, gum arabic, etc., toward copper sulphate and sodium hydroxide. The test was devised by Liebermann¹⁴ for detecting dextrin and gum senegal in the presence of gum arabic. The precipitate formed in a solution of gum arabic by adding copper sulphate and then an excess of sodium hydroxide, balls together and rises to the top. That from senegal is less in amount and is in finer particles that remain distributed throughout the liquid. On warming, these precipitates dissolve to only a slight extent, and even on boiling there is no reduction to cuprous oxide. A solution of dextrin yields a precipitate that dissolves on warming. The cupric salt contained in the dark-blue liquid is completely reduced by boiling.

To these statements of Liebermann we may add that gum ghatti gives a dark-blue solution which is, at times, slightly turbid, but clears up on warming gently. There is slight reduction on boiling, but this may not be noticed unless a long time is allowed for the cuprous oxide to settle.

In the second part of this paper is described a quantitative method for gum arabic, based on a modification of Liebermann's reagent.

6. MISCELLANEOUS QUALITATIVE TESTS.—Many other reagents for gum arabic have been suggested from time to time, but most of them deserve only brief mention.

One of the most interesting of these depends on the presence of an oxidase in gum arabic, and was devised for the detection of this gum when mixed with tragacanth, in which there is no such

¹² Rideal and Youle: *J. Soc. Chem. Ind.*, **10**, p. 610; 1891.

Fromm: *Z. anal. Chem.*, **40**, p. 143; 1901.

Papasogli: *L'Orosi*, **21**, pp. 263-265; 1898.

Scheibler: *Z. Ver. d. Zuckerind.*, **23**, p. 288.

Battut: *Sucr. Indig. et Colon.*, **32**, p. 285.

v. Lippmann: *Chemie der Zuckerarten*, p. 1616.

Allen: *Coml. Org. Anal.*, 4th ed., **1**, pp. 440 et seq.

¹³ "Zündwaren" in Lunge: *Chem.-Techn. Untersuchungsmeth.*, 6th ed., **3**, pp. 167-168.

¹⁴ *Chem.-Ztg.*, **14**, p. 665; 1890.

enzyme.¹⁵ To a suspension of the powdered gum in 30 parts of cold water is added an equal volume of a 1 per cent aqueous solution of guaiacol, followed by a drop of hydrogen peroxide. If gum arabic is present, the mixture at once turns brown. Dextrin, of course, does not react. Gum ghatti may not react, even after standing for half an hour. With some pieces, however, the color is produced. This recalls the behavior of the gum toward a mixture of alcohol and hydrochloric acid. (See under 3, above.)

Bornträger¹⁶ suggested warming a suspected sample with 50 per cent hydrofluoric acid. When much bone glue is present, the odor of butyric acid is noticed. Dextrin is detected by its own characteristic odor, while with gum arabic only that of hydrofluoric acid is noticed.

According to Petri,¹⁷ aliphatic aldehydes, certain sugars, and gum arabic give a fuchsin-red color when mixed with alkali and diazobenzenesulphonic acid. The color is gradually developed.

Günsberg¹⁸ used a fresh solution of egg albumen, acidified with acetic or hydrochloric acid, as a reagent for gum arabic. Dextrin yields a dense precipitate which is insoluble in an excess of acid or of dextrin solution. With gum arabic only a little must be added, for the precipitate first thrown out dissolves in even a slight excess. Any albumen that is not precipitated by the gum arabic can be coagulated by heat, although under ordinary circumstances acidified albumen is not coagulable.

Vamvakas¹⁹ found that as little as 5 per cent of gelatin can be detected in gum-arabic mucilage by means of Nessler's reagent. The mucilage is shaken with one-fourth its volume of the reagent and allowed to stand. If no gelatin is present, the brownish-gray precipitate remains suspended for some days. With an admixture of gelatin the precipitate is dull gray and settles more rapidly. In the presence of tartaric acid even a 30 per cent solution of gum arabic yields but a slight precipitate with Nessler's reagent.

¹⁵ Payet: *Apoth. Ztg.*, 25, p. 116; *Z. anal. Chem.*, 44, p. 453; 1905.

¹⁶ *Oesterr. Chem. Ztg.*, 3, p. 1888; *Z. anal. Chem.*, 40, p. 131; 1901.

¹⁷ *Z. physiol. Chem.*, 8, p. 291; *Jahresber.*, 1884, p. 1328.

¹⁸ *J. prakt. Chem.*, 88, p. 239; *Z. anal. Chem.*, 2, p. 218; 1863.

¹⁹ *Ann. Chim. Anal. Appl.*, 12, pp. 12 and 139; 1907. *Analyst*, 32, pp. 93 and 226; 1907.

These reactions call to mind the mixture of mercuric acetate and sodium chloride suggested by Hager.²⁰ When warmed with glucose there is no change, while gum arabic, dextrin, and cane sugar reduce the mercuric salt with formation of calomel.

In this connection also should be mentioned the reduction of molybdic acid when boiled with a solution of dextrin.²¹ A blue solution results, while with gum arabic there is no change.

Analogous to the reaction with copper sulphate and alkali is the formation of a stable blue color when a solution of gum arabic or of dextrin is mixed with cobalt-nitrate solution, followed by dilute sodium hydroxide.²²

Zsigmondy,²³ using a deep red solution of colloidal gold as a reagent for colloids, studied the color changes produced by glue, gums, albumen, etc.

Other reagents for gum arabic, such as sodium or potassium silicate, borax, or ammonium oxalate,²⁴ would seem to depend largely upon the natural calcium content of the gum, though some of the reagents are said to produce gelatinization.

In our work solutions of various salts were mixed with 2 per cent solutions of gum arabic, but in no case was there precipitation or gelatinization. These salts need not be enumerated, except to say that, among others, such promising reagents as basic zinc salts and sodium zincate were found to be useless.

Gum arabic gives no color reaction with iodine solution.

7. CHIEF QUALITATIVE TESTS FOR GUMS AND DEXTRIN.—

Most of the tests described above, as well as others not mentioned, were tried. Most of them were unsatisfactory. After the work had progressed far enough to show which tests are reliable we succeeded in obtaining new samples. The five or six lots already on hand were from different dealers and the original sources were not known. The new ones included gum arabic from Khartum and Kordofan, a second supply of gum ghatti, and the closely related gum gedda. It is unnecessary to discuss these in

²⁰ Pharm. Centralhalle, 18, p. 313. Z. anal. Chem., 17, p. 380; 1878.

²¹ Hager: Commentar zur ersten deutschen Pharmakopöe, II, p. 116. Cf. Stohmann in Muspratt, 4th ed., 3, p. 1917; 1891.

²² Papasogli: I'Orosi, 21, pp. 263-265; 1898. Chem. Centr., 69, II, p. 991; 1898.

²³ Z. anal. Chem., 40, p. 697; 1901.

²⁴ Allen: Loc. cit., p. 441.

detail since their reactions are included in the table below. All samples of gum arabic, from whatever source, behaved alike. Gum gedda, it will be seen, acts much like a mixture of gums arabic and ghatti. Only the tests that are most reliable are included. The gum solutions were prepared by dissolving 1 g. of the solid in 50 cc of hot water and straining through a loose plug of cotton.

TABLE 1
Chief Qualitative Tests for Gums and Dextrin

Reagent	Gum arabic	Gum ghatti	Gum gedda	Dextrin
Ferric chloride and alcohol.	Precipitate.....	Nearly clear.....	Slight precipitate....	Slightly cloudy.
Basic lead acetate....	Dense precipitate..	Cloudy.....	Less precipitate than arabic.	Slightly cloudy.
Copper sulphate and sodium hydroxide (cold).	Blue precipitate, colorless solution.	Dark - blue solution, often cloudy.	Light-blue precipitate, dark-blue solution.	Dark-blue solution.
As above, but boiled..	Precipitate darker, pale-blue solution.	Slight reduction...	Precipitate darker...	Complete reduction.
Iodine solution.....	(a)	(a)	(a)	Reddish-purple solution.

^a No characteristic color reactions.

III. QUANTITATIVE METHODS

Since the chemical nature of the gums is such as to render most of the qualitative tests of at least doubtful value, it is not surprising that there are difficulties involved in their quantitative separation and determination. Some of the methods that have been proposed will be reviewed below, following which our own procedure will be described.

1. FERRIC CHLORIDE AND CALCIUM CARBONATE.—The first qualitative test mentioned above was the formation of a precipitate when neutral ferric-chloride solution and alcohol are added to a solution of gum arabic. This precipitate is soluble in dilute acid, so that the gum is not completely thrown down unless the acid that is simultaneously set free is neutralized. Roussin²⁵ found that calcium carbonate is satisfactory for the purpose. The gum solution is mixed with alcohol and ferric chloride, and

²⁵ Loc. cit. See also Allen; loc. cit.

an excess of the carbonate is then added. The mixture of precipitated gum and ferric hydroxide, which is rather difficult to filter, is washed with alcohol, dissolved in hydrochloric acid, the gum thrown out by strong alcohol, washed with the same reagent, dried, and weighed.

Roussin devised this method for the quantitative separation of gum arabic from dextrin. It is recommended by Allen, and was found by Auguet to give good results in determining the gum in sirups.²⁶

2. FERRIC CHLORIDE AND SODIUM CARBONATE.—Another method based upon the reaction with ferric chloride was suggested by Landwehr²⁷ for the determination of glycogen and, incidentally, of gum arabic. Concentrated ferric-chloride solution is added in slight excess, followed by concentrated sodium carbonate until all of the iron is thrown down. The precipitate is at once filtered off, washed with hot water, dried at 110°–120°, and weighed. It is then ignited and the weight of ash deducted from that of the precipitate.

Determinations were made of the percentages of water held by ferric hydroxide when dried to constant weight at different temperatures. A correction for this water, calculated from the excess of ferric chloride, is applied to the weight of the original precipitate. It is not clear, however, just how this excess is to be determined, nor is the retention of moisture by the precipitated gum taken into account.

3. LEAD ACETATE.—As already stated, Chauvin²⁸ suggested the employment of an alcoholic solution of neutral lead acetate for the precipitation of gum, especially from sirups, but found that long standing was necessary. Rocques and Sellier²⁹ also found that this procedure gives good results.

As far as we are aware, the basic acetate has not been used for the quantitative determination of gum. Various formulas have been ascribed to the precipitate,³⁰ and it is not unlikely that it may vary in composition.

²⁶ *Ann. Falsific.*, 2, pp. 136–163; *Chem. Abstr.*, 5, p. 538, 1911.

²⁷ *Z. physiol. Chem.*, 9, p. 164, 1884.

²⁸ Footnote 9; see also *Ann. Falsific.*, 5, pp. 27–30; *Chem. Zentr.*, 83, I, p. 756; 1912.

²⁹ *Ann. Chim. Anal. Appl.*, 16, pp. 218–220; 1911; *Chem. Zentr.*, 82, II, p. 394; 1911.

³⁰ Riegel: *Arch. Pharm.* [2], 54, p. 155; *Jahresber.*, 1, p. 795; 1847–48.

Battut: *Sucr. Indig. et Colon.*, 32, p. 285.

Scheibler: *Z. Ver. d. Zuckerind.*, 23, p. 283.

4. ALCOHOL.—Chauvin,³¹ on account of the slowness of precipitation when lead acetate is used, employed alcohol acidified with hydrochloric acid. The addition of the latter is not necessary, for the gums are all insoluble in strong alcohol. This has often been taken advantage of to remove them from wines, plant extracts, etc.³²

5. MISCELLANEOUS METHODS.—Gum arabic can be hydrolyzed to a mixture of hexoses and pentoses by warming with dilute mineral acid, though not all samples yield these in the same proportions. Hence, quantitative methods based upon the use of Fehling's solution (hexoses), or upon the estimation of furof (pentosans) have failed.³³

By digesting with moderately strong nitric acid, gum arabic is oxidized to mucic acid, which can be determined with fair accuracy, but samples of gum from different sources yield different percentages of the oxidation product.³⁴

Williams,³⁵ working with quite a number of gums and resins, determined the iodine absorption number and the acidity of gum arabic, as well as the amount of alkali taken up when it is boiled with alcoholic potash. His figures showed that samples of gum differ so widely that methods involving these determinations are of no value.

6. COPPER SULPHATE AND ALKALI.—Madsen³⁶ estimated the gum in licorice juice (*succus liquiritiae*) by throwing it out with strong alcohol, dissolving in a little water, and then treating with a 10 per cent solution of copper sulphate, followed by an excess of sodium carbonate. The precipitate is probably identical with that formed by the combined action of copper sulphate and sodium hydroxide. After filtering off the precipitate, it is washed with weaker and weaker soda solution, dissolved in dilute hydrochloric acid, and the gum thrown down by 85 per cent alcohol. After washing with alcohol it is dried and weighed. The weight, multiplied by 1.21, gives the percentage of gum. This is not said to

³¹ Loc. cit.

³² Diehl: *Pharm. Rundschau*, 1, p. 31; *Z. anal. Chem.*, 22, p. 622; 1883.

Madsen: *Pharm. Centralhalle*, 20, p. 144; *Z. anal. Chem.*, 22, p. 134; 1883.

³³ v. Lippmann: Loc. cit.

³⁴ Kiliani: *Ber. d. Chem. Ges.*, 15, pp. 34-37; 1882.

³⁵ *Chem. News*, 58, p. 224; *Z. anal. Chem.*, 28, p. 732; 1889.

³⁶ Loc. cit. See footnote 32.

be identical with gum arabic, but for botanical reasons may well be quite similar to it.

During the course of our tentative experiments, and without knowledge of the work of Madsen, an attempt was made to precipitate gum arabic by means of copper sulphate (5 g of the crystallized salt in 100 cc of water) and sodium carbonate (13.25 g of the anhydrous salt in 100 cc of alcohol of the maximum strength that would give a clear solution). Whether the soda was added before, or after the copper solution made no difference in the result. The filtrate was always cloudy, even though the precipitate was so gelatinous that filtration was very slow.

Promising, though still too low, results were obtained with 10 per cent sodium hydroxide, instead of soda, followed by the addition of enough absolute alcohol to bring the total up to about 60 per cent strength. The precipitate was washed with alcohol, dried, weighed, and ignited. After deducting the weight of the ash, the percentages of gum found in four determinations varied from 84.6 to 90.1. In each of these four determinations the weight of ash from the copper precipitate was a little greater than that of the gum taken, owing to the fact that there was a considerable excess of copper in the form of hydroxide. Since this increased the time required for drying, besides being otherwise objectionable, some qualitative tests were made with a modified Fehling solution, with the hope that these difficulties would be avoided. Twenty cubic centimeters of copper-sulphate solution and 15 cc of sodium-hydroxide solution, of the strengths stated above, were mixed, enough Rochelle salt was added to dissolve the precipitate, and the whole diluted to 100 cc. To a solution of about 0.25 g of gum in 40 cc of water there was added 50 cc of the Fehling solution, but with no apparent change, except at times a slight cloudiness. While stirring vigorously, 90 cc of 95 per cent alcohol was then added, whereupon the familiar blue precipitate formed in fine particles that soon settled. Blanks showed that no cloudiness was produced in the Fehling solution when mixed with water and alcohol in the quantities stated.

The precipitate was filtered off, washed successively with 50, 75, and 95 per cent alcohol, dried at 110° to 115°, weighed, etc., as before. Two determinations, made exactly as stated, gave the

values 95.6 and 94.9 per cent of gum. When the gum was dissolved in 25, instead of 40 cc of water, the percentage found was 97.2. The original dried gum yielded 2.8 per cent of ash, chiefly calcium carbonate, with a little potassium carbonate. No correction was made for this ash in calculating the above percentages. In each of the three determinations just mentioned the weight of the ash from the copper precipitate weighed less than half as much as the gum.

Since the gum-copper precipitate is of a gummy nature, it is not possible to completely remove foreign salts by any amount of washing. Both copper sulphate and sodium sulphate are insoluble in alcohol of the strength used. This is not true of the respective acetates or of Rochelle salt, so that in another series of determinations copper acetate was employed. It is not necessary to go into details further than to say that the results were a few per cent below or above 100 with varying dilution of the gum solution and of the Fehling solution, and according to the final percentage of alcohol in the mixture.

Even this reagent was not ideal, for there must always be present the excess of copper salt, as well as the Rochelle salt and the sodium hydroxide and acetate.

7. METHOD FINALLY ADOPTED.—Copper acetate dissolved in ammonia possesses obvious advantages over all the other solutions employed. In order to ascertain the best conditions for precipitation, a few preliminary tests were made. One-quarter gram of gum arabic was dissolved in 50 cc of water, 25 cc of ammoniacal copper-acetate solution (see below) added, and then enough alcohol to give the required percentage of the total volume of the mixture. Thirty per cent of alcohol gave no precipitate. With 40 per cent there was a faint precipitate, so finely divided that it was practically impossible to filter. With 50, 60, and 70 per cent of alcohol, respectively, dense and easily filtered precipitates were formed. The addition of more alcohol to the filtrates from these three caused no further precipitation. These precipitates gave on ignition the following amounts of ash, practically cupric oxide.

TABLE 2

Ash Found With Varying Alcohol Content

Alcohol.....	50%	60%	70%
	G	G	G
Ash.....	0.0412	0.0436	0.0544
	.0402	.0446	.0564
	.0398	.0452	.0548
Average.....	.0404	.0445	.0552

From this it will be seen that in the presence of 50 per cent of alcohol the gum is precipitated quantitatively and carries down with it less occluded matter than when the mixture contains more alcohol.

The following procedure was finally adopted for the determination of gum arabic: Fifty grams of copper acetate was dissolved in water, an excess of ammonia added, and the solution diluted to 1000 cc, using water and alcohol in such proportions that the final solution contained 50 per cent of alcohol. For each determination a 50 cc portion of a gum-arabic solution, representing 0.25 g of gum, was pipetted into a 250 cc beaker, an equal volume of alcohol added, and then 25 cc of the copper reagent, with constant stirring. The precipitate was allowed to settle, was filtered on a tared paper, washed with 50 per cent alcohol containing ammonia, then with 75 per cent, and finally with 95 per cent alcohol. It was dried to constant weight at 105°, ignited in a porcelain crucible, and the ash weighed. The amount of ash was deducted from the weight of the original precipitate and the difference called "net gum arabic." The amount of moisture in the gum originally taken for analysis must be allowed for. This is determined by drying in a current of hydrogen at 105°. No allowance is made for the potassium and calcium which form an integral part of the gum. These may be to some extent retained in the precipitate and, therefore, be included in the ash. Any error that may be introduced by neglecting this is small and very much less than the error inherent in the method.

In some of the preliminary work the gum-copper precipitate was dried at 95° and then at 105°. The additional loss in weight

at the higher temperature was usually about 3 or 4 mg, or 1.5 per cent.

The following table gives the results of a number of determinations on 0.25 g portions of gum. Since this contained 11.5 per cent of moisture, the amount of actual gum was 0.2213 g. The precipitates were dried at 105°.

TABLE 3
Results Obtained By the Ammonia-Copper Acetate Method

Series	Total precipitate	Ash	Net gum	Percentage found	Average of series
	G	G	G		
A.....	0.2570	0.0433	0.2137	96.6	97.3
	.2596	.0428	.2168	98.0	
	.2613	.0470	.2143	96.9	
	.2647	.0469	.2178	98.4	
	.2554	.0413	.2141	96.7	
B.....	.2970	.0755	.2215	100.1	99.5
	.2840	.0649	.2191	99.0	
	.2837	.0663	.2174	98.3	
	.2887	.0689	.2198	99.3	
	.3012	.0785	.2227	100.7	
C.....	.2880	.0659	.2221	100.4	99.9
	.2908	.0668	.2240	101.2	
	.2844	.0651	.2193	99.1	
	.2853	.0646	.2207	99.7	
	.2827	.0640	.2187	98.8	
D.....	.2880	.0633	.2247	101.6	102.6
	.2927	.0653	.2274	102.8	
	.2982	.0697	.2285	103.3	
General average.....				99.5	

It is evident from an inspection of the figures that the method as finally modified is capable of yielding results as accurate as can be expected. It is realized, however, that much work could yet be done with mixtures of gum arabic with ghatti, dextrin, etc., as well as with mucilages of known composition. A few preliminary determinations, made while Fehling solution was still being experimented with, indicate that dextrin and ghatti tend to be carried down with the gum arabic. At the same time a sample of mucilage was prepared according to the formula in the United States Pharmacopoeia; from this the gum was precipitated by strong alcohol, dried, and analyzed. The net gum found in four deter-

minations was about 91 per cent. This low result may be due to a fault of the method or to partial hydrolysis of the gum.

The publication of the results thus far obtained has been so long delayed, and the opportunity to complete the work seems so remote, that it has been decided to present this paper without further postponement.

IV. SUMMARY

An incorrect report to the effect that a certain sample of mucilage contained no gum arabic led to a study of many of the published tests for this gum, as well as to a search for others than the few that proved to be reliable. It was found that basic lead acetate gives the most characteristic reaction, while mixtures of copper sulphate and sodium hydroxide, and of neutral ferric chloride and alcohol are of value as confirmatory tests.

A summary of the more important methods that have been proposed for the quantitative estimation of gum arabic is next given, followed by a description of the steps that led the authors to the use of alcoholic copper acetate-ammonia solution for this determination.

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